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Article

Comprehensive and Sustainable Recycling Process for Blended Different Types of End-of-Life Solar Panels: Leaching and Recovery of Valuable Base and Precious Metals

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Abstract: The production of photovoltaic modules is increasing to reduce greenhouse gas emissions. However, this results in a significant amount of waste at the end of their lifespan. Therefore, recycling these solar panels is important for environmental and economic reasons. However, collecting and separating Crystalline silicon, Cadmium Telluride, and Copper indium gallium selenide panels can be challenging, especially in underdeveloped countries. The innovation of this work is to develop a process to recycle all solar panel waste. The dissolution of all metals is studied through the leaching process as the main step of the flowchart. In the first step of leaching, silver can be recovered 98% by 0.5 nitric acid. Then, the second and third step involves the use of glycine for base metals dissolution, followed by the leaching of valuable metals with hydrochloric acid. The effect of parameters such as initial pH, acid concentration, solid/liquid ratio, and Hydrogen peroxide concentration are studied. The results show that Cu, Pb, Sn, Zn, Cd, In, Ga, and Se can be recovered until about 100% under optimal conditions. The optimal conditions for the dissolution of Cu, Zn, and Cd were the glycine concentration of 0.5 M, Temperature of 25 °C, the solid/liquid ratio of 10 gr/l, and 1% of Hydrogen peroxide. The optimized glycine concentration for the leaching of lead and tin was 1.5M. Indium and gallium were recovered to 100% by the use of 5M Hydrochloric acid, S/L=10gr/l, and T=45 °C. Separation of selenium and tellurium occurred by 0.5 M HCl at a temperature of 60 °C. Additionally, for the first time, a general outlook for the recycling of various end-of-life solar panels is suggested.

Keywords solar panel recycling; leaching; base metals; precious metals; waste management; environmental impact; sustainable technology

1. Introduction

In recent years, solar panels have played a significant role in reducing global warming by generating clean and emission-free electricity from the sun, thus reducing reliance on greenhouse-gas-producing fossil fuels [1]. Solar energy is considered the fuel of the future due to its potential for meeting increasing electricity demand and reducing greenhouse gas emissions. As a result, the mass production of solar panels using different technologies has increased in recent decades, and the production of new generations of solar panels is expected to continue [1,2]. However, despite the positive impact of solar panels, their production also has negative consequences, including the generation of large amounts of waste. The average lifetime of a PV panel is 25 years, and given their worldwide production, it is anticipated that there will be a significant amount of waste generated annually. According to the International Renewable Energy Agency (IRENA) report, apart from considering the generation of a large amount of waste during the manufacturing process of solar cells [3,4], an estimated 80 million tons of PV waste will be generated by 2050 [5].

The issue of solar panel waste is significant from two perspectives. Firstly, these wastes contain lead, cadmium, and other harmful chemicals that can cause significant health and environmental

hazards [6,7]. Secondly, these wastes are considered valuable due to their high content of valuable metals [7–10]. Therefore, the life cycle assessment (LCA) should be applied to evaluate all aspects of the environmental impacts, energy consumption and production, and emissions during the entire life cycle of solar panel technology. LCA is a feasible method that can be used as an environmental management tool to consider the positive ecological effects of solar panels due to the decrease in carbon emissions and energy consumption. This should also evaluate the potential cradle-to-grave life cycle impacts of solar panels after their service life, as it is uncertain what will happen to this massive amount of solar panel waste.

Moreover, in addition to the issue of waste management, the recovery of metals from these wastes and their reuse should also be considered. Although few LCA studies have explored the recycling of PV technologies, some have investigated the production and use of PV technologies [11,12], and energy consumption due to PV recycling [13]. The main factors affecting end-of-life panels' waste management are self-take-back collection, recycling facilities, and material recovery [14].

The most commonly used PV panels are crystalline silicon and thin-film PV cells. The former accounts for around 80% of the market share, but this is decreasing due to the increased capacity and the shortage of production costs [15]. Conversely, the lower production costs and optimum efficiency of thin-film panels are driving their growth in the overall photovoltaic market [15–17]. However, the handling and waste management of these extensive ranges of used solar panels that contain Cd, Se, Pb, and other environmentally hazardous metals pose an environmental concern. Additionally, there is another concern about the fate of these sources of valuable metals. These two perspectives will force industries and governments to plan for PV waste safe disposal or recycling shortly.

Although many researchers have studied the recycling processes of used solar panels [18–22], only two processes have been established on an industrial scale. The recycling of C-Si and CdTe thin-film modules is operated by Deutsche Solar and First Solar, respectively [23–25]. The first step in the recycling process is the separation of the modules, followed by the separation of the non-metal and metal parts. Many separation methods have been developed for C-Si [26–29], CdTe [30–32], and Copper-indium-gallium-selenide (CIGS) [18,33,34], most of which include physical, mechanical, and chemical processes. This step involves the elimination of glass, Al, and plastic, as well as the separation of metal and non-metal parts from solar panels. The second recycling processes are different for different types of solar panels. The recycling processes for silicone solar modules typically involve delamination and metal extraction. The solar cell electrodes and interconnected ribbons, made of silver, aluminum, and copper, are dissolved into aqueous media for recycling [35,36]. Two of the most advanced processes developed worldwide are the Full Recovery End-of-Life Photovoltaic (FRELPA) and Baseline processes [37].

Recovery of cadmium and tellurium from cadmium telluride PVs is difficult due to the low content of the semiconductor [38]. There are many hydrometallurgical recycling processes for CdTe as well as acid dissolution and subsequent precipitation [39], cementation [40] electroplating [41], and ion exchange [42,43]. A recycling process for CdTe PVs based on a sequence of mechanical steps rather than wet-chemical techniques has also been proposed [44].

In recent years, researchers have shown significant interest in end-of-life CIGS panels due to the presence of gallium and indium [45,46]. Several recycling processes such as mechanical techniques [47], wet chemical process [48], electrochemical method [49], and leaching and electrolyzing of metals have been developed [50]. Xiang Li and colleagues have reported an effective separation process by an alkaline agent [51]. They found selective alkali leaching is feasible to separate indium and gallium effectively as pure In_2O_3 and Ga_2O_3 separately. The recycling of copper, indium, and gallium from thin-film solar panels has also been reported [52], where the H_2SO_4 is used as a leaching agent. In many researchers, the separation of indium and gallium was studied by solvent extraction and ion exchange following the leaching step [53].

On the topic of recycling, there is an important point that using a single treatment or recycling process is not usually practical for recycling all waste of a particular type [54]. Given the variety of solar panels on the market and the need for economically feasible recycling processes, an

investigation of a general procedure for the extraction of metals from the metallic parts of various solar panels is suggested in this work. The recovery of metals through the co-processing of all types of solar panels was examined. To do this, the metallic and non-metallic parts from Si, CdTe, and CIGS solar panels were separated and subjected to hydrometallurgical processes. This section is the initial step of the suggested flowsheet and includes the separation of metallic and non-metallic parts and comprehensive leaching studies of copper and other base metals. Copper is the main component of solar PV systems due to its thermal and electrical conductivity, and other base metals are also used in solar PV systems. Many hydrometallurgical methods have been studied for the dissolution of copper and other base metals; most of these studies used two or more leaching agents in two or more stages [55–59]. The simultaneous leaching of copper and solder alloy was studied by a new method from PCB waste with HBF_4 as a leaching agent [60]. After the leaching step, there were different methods for the separation of copper from other metals including solvent extraction [60–64], precipitation [65,66], and cementation [67]. In the second step, the recovery of gallium and indium by chemical leaching has been proposed. The optimum conditions for the selective extraction of copper, the further leaching step of other base metals, and the extraction of indium and gallium have been investigated.

The study presented in this paper proposes a comprehensive and innovative approach for the extraction of valuable metals through the co-processing of all types of solar panels, which is a significant contribution to the field of solar panel recycling. By utilizing glycine, an eco-friendly leaching agent, in the initial step of the process, this recycling method is not only environmentally friendly but also economically feasible. In many studies, it was proven that the leaching of base metals has been successfully conducted by glycine as a dissolution agent [68–71]. The study also provides insights into the leaching behavior of various metals under different conditions, allowing for the selective extraction of different metals from solar panels. Furthermore, the paper presents a general flowsheet for the recycling of all types of solar panels, which has the potential to be implemented globally in the future, considering its economic and environmental benefits. The study highlights the importance of developing comprehensive recycling methods for spent solar panels to ensure the effectiveness of solar PV technology and reduce its environmental impact. This approach appears to be a significant contribution to the field of solar panel recycling, and it could potentially pave the way for future research and development in this area.

2. Materials and Methods

2.1. Material and Reagents

To conduct the research, a variety of C-Si, CIGS, and CdTe solar panels, including alloy layers such as CdTe and CIGS, were collected. While thin-film solar panel technology has not yet been established in Iran, the collection of these materials allowed for the development of a new route for recycling all kinds of solar panels in a single plant. The metallic parts were separated from other parts through physical processing and thermal treatment [32], making it economically and environmentally feasible to recycle all metals from different types of photovoltaic panels through the same continuous process route. Analytical-grade nitric acid (65%) for the leaching of silver, glycine (50%) for the leaching of base metals, hydrochloric acid (37%) for the valuable metals leaching, hydrogen peroxide (30 wt.%) as oxidant and ammonia (25%) for the increasing of pH were purchased from Merck Chemicals Company. All aqueous solutions were prepared using distilled water.

2.2. Initials Preparation of Samples from Different Types of Solar Panels

Although, the compound of solar panels can be different due to the different manufacturers and rapid changes in the innovations of technology. The main goal of this study is to find a novel route for recycling all types of PV panels. Since the recycling of these PV panels from the point of view of LCA has been studied, considering that all metals and non-metals parts should be recovered as much as possible. to achieve this goal, the common multi-layered structure of all PV panels in the preparation step, including glass and polymeric matrix (EVA, PVF, or Tedlar) have been considered.

The preparation step for different kinds of PV panels begins with the shredding process after the manual dismantling of the Al frame. This step is followed by gravitational separation, magnetic separation, and thermal treatment according to previous research [22,27,30,33]. The PV panels were cut into small pieces of 10 *10 cm via a cutting machine. These pieces were shredded by an SM-2000 cutting mill (Retsch, Germany) and sieved into different fractions using standard sieves. The majority of glass was recovered in a fraction of >1mm. Also, the EVA was completely removed after thermal treatment at 700 °C [72,73]. The solid feed material in the leaching process is ≤ 1 mm. As it is shown in Table 1, this sample includes many components including BMs, PMs, and other valuable metals.

Table 1. The XRF results of major metal contents of the sample (Prepared in this study by using end-of-life solar panels and alloy layers).

	Cu	Pb	Sn	Cd	Zn	Ag	Se	In	Ga	Te
Metal content (Wt%)	38	1.9	1.1	0.81	5.9	0.56	0.12	0.09	0.08	0.51

2.3. Methods

All leaching experiments were carried out in a 1L autoclave equipped with a mechanical stirrer, a reaction temperature-control unit (298-313K (25–80°C)), and a condenser to avoid loss of solution (Figure 1). The initial pH value was adjusted to a preset value by carefully adding ammonia solution, and the acidity of the solution was measured using a Mettler Toledo SevenExcellence S400 pH Meter. The agitation was fixed at 300 rpm. At the end of the leaching time, the slurry was filtrated and the obtained filtrate was delivered to the analyzing step.

In the first step, diluted nitric acid was used to leach the samples and recover silver. The goal of this step is the selective extraction of silver over other metals. Many researchers found that HNO_3 has a good performance in the selective dissolution of silver over tin and lead [27,74–76]. After leaching, the samples were dried, and subsequently, leaching experiments were conducted with glycine. To determine their impact on the leaching efficiency of base metals, the effects of glycine concentration, initial pH value, H_2O_2 volume concentration, time, and liquid-to-solid ratio were evaluated. The experimental conditions were based on the potential-pH diagrams of the copper-glycine system at 25°C and 1 atm, as described in a study by Serdar Aksu and Fiona M. Doyle [77]. After glycine leaching, the raffinate was subjected to the next experiments. The residue solid was prepared for the next leaching step by roasting as the feed material. In the final leaching step, HCl was used as the leaching agent, and the raffinate from the leaching step was analyzed for the measuring of the extraction rate. The effect of liquid-to-solid ratio, the acid concentration, temperature, and pH Was studied as the affecting parameters on the second leaching step.

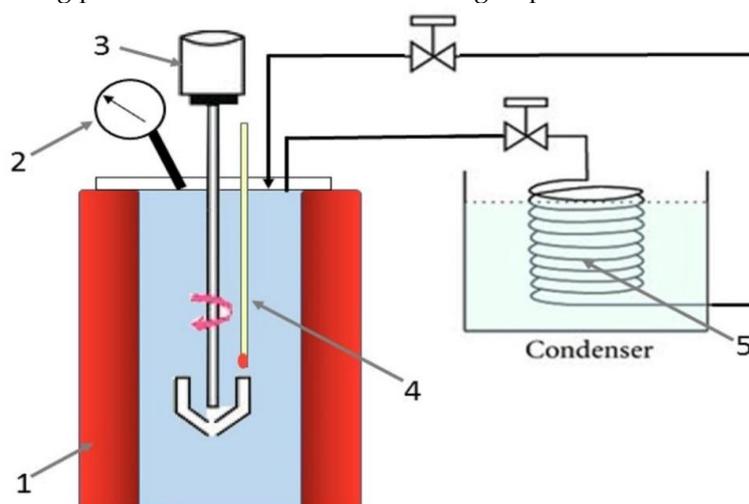


Figure 1. Schematic diagram of the lab-scale autoclave reactor 1-Electric heater, 2-Pressure sensor, 3-Stirrer, 4- Temperature sensor, 5- Condenser.

2.4. Multivariate Design of Experiment

The aim of this study is the development of a complete and innovative flowsheet for the co-processing of all types of solar panels. To achieve that, the procedure of leaching optimization of the operating parameters via the response surface methodology (RSM) is employed. A Box-Behnken Design was utilized to investigate the influence of four factors on the response of Recovery of Copper, Zinc, Cadmium, Lead and Tin respectively. The study comprised of 28 experimental runs using the Response Surface Methodology (RSM) and Design Expert 13. The design includes one block and four central points for each block. The numerical factors considered in the study were Glycine acid concentration, solid/liquid ratio, initial pH and H₂O₂ dosage, between -1 and +1, as shown in Table 2.

Table 2. Factors and their levels.

Factor	Name	Unit	Minimum	Maximum	Coded Low	Coded High
A	[GLY]	M	0.10	1.50	-1 \leftrightarrow 0.10	+1 \leftrightarrow 1.50
B	S/L	gr/l	5.0	200	-1 \leftrightarrow 5.0	+1 \leftrightarrow 200
C	pH		8.0	13.0	-1 \leftrightarrow 8.0	+1 \leftrightarrow 13.0
D	H ₂ O ₂	%	0.0	1.0	-1 \leftrightarrow 0.0	+1 \leftrightarrow 1.0

The quadratic polynomial regression model (Equation (1)) was used to predict the response behavior while varying the four independent variables:

$$R = a_0 + \sum_{i=1}^4 a_i Y_i + \sum_{i=1}^4 a_{ii} Y_{ii}^2 + \sum_{i=1}^3 \sum_{j=i+1}^4 a_{ij} Y_i Y_j \quad (1)$$

where R represents the response, including copper recovery percentage and Cu concentration. The intercept, linear, quadratic, and interaction coefficients are denoted by a_0 , a_i , a_{ii} , and a_{ij} , respectively. The four independent variables, namely Glycine acid concentration, solid/liquid ratio, initial pH and H₂O₂ dosage, are represented by Y_i and Y_j . After achieving the quadratic polynomial model, analysis of variance (ANOVA) was applied to validate the provided model. Due to the complicated relationship of parameters, their fluctuating behavior was studied and explained individually.

2.5. Analytical Procedure

Inductively coupled plasma (ICP PlasmaQuant 9100 Series) was used for the analysis of the leached metals present in the leach liquor. The metals leaching rate was expressed as the dissolved percentage of metals which was measured by the difference between the value of dissolved metals and the initial amounts. The mineralogical phase analysis was carried out using an X-ray diffractometer (XRD, EQUINOX3000). An X-ray fluorescence spectrometry (XRF, PW2400) was used for multi-element analysis.

3. Results and Discussion

3.1. The Leaching Behavior of Base Metals

Generally, the essential role of the leaching step as the basis of most hydrometallurgical extraction processes is getting the maximum amount of the metals. But also, the aim is to find a selective, low temperature, and low chemical consumption process for metals. If there are many metallic components in a solid matrix, the selectivity of a leaching system can be obtained by the chemical affinity of the component between the reagent and the kinetic consideration. Even though

the first strategy is based on the differences in affinity of a given reagent for reacting with the various components, the second strategy is based on the different component's dissolution rates.

In this step, the samples were leached in diluted nitric acid to recover silver [27], and a 98% percentage of silver was leached. Silver is used on the surface of panels, the 0.5 M diluted acid dissolved 98% silver and less than 5% of lead, copper, zinc, and tin. However, these metals were more active than silver, but because of the 15 min of leaching time and the 0.5 M acid concentration, only very small amounts of these metals dissolved.

Following the elimination of silver, Figure 2 shows the dissolution rate of metallic components from end-of-life solar panels by glycine acid. The condition of the experiments was considered at $T=25^{\circ}\text{C}$, $[\text{GLY}]=0.5\text{ mol/l}$, and $\text{S/L ratio}=20\text{gr/l}$. As shown in Figure 2, the dissolution of In, Ga, Te, and Se was negligible in comparison with other metals. It can be predicted that the glycine can dissolve copper easily, but the glycine dissolved zinc and cadmium up to the ranges of 87% and 64% under given conditions. As it is clear, glycine forms the soluble complex with copper, cadmium, lead, and zinc. But the stability constant of $\log K$ is different for these metals. As reported in the research, the $\log K$ of glycine complexes with Cu is 8.56 (at $I=0$) and this value is higher than the $\log K$ for Cd, Zn, and Pb, 4.7, 5.38, and, 5.47, respectively [78,79]. As it is shown in Fig 2, the dissolution of zinc is sharper and higher than cadmium. The standard reduction potentials of zinc, cadmium, and copper are -0.76 V , -0.4 V , and 0.34 V respectively [80].

regarding the different dissolution percentages of Zn and Cd, the probable reaction which can occur is cementation:



It is probable for the dissolved Cd to be cemented by undissolved Zn, leading to a higher percentage of Zn dissolution compared to Cd, especially at the initial stages of the leaching process. Due to the standard reduction potentials of metals, the cementation of cadmium happened through the presence of few amounts of solid zinc [81,82].

When 50% of zinc was dissolved, the dissolution of cadmium suddenly increased. whereas, the dissolution of zinc and copper occurred simultaneously at the beginning of leaching, and after that, the dissolution of copper was very faster than zinc. As shown in Figure 2, Glycine could not dissolve In, Ga, Se, and Te under these conditions, and the dissolution of tin was very low compared to other base metals.

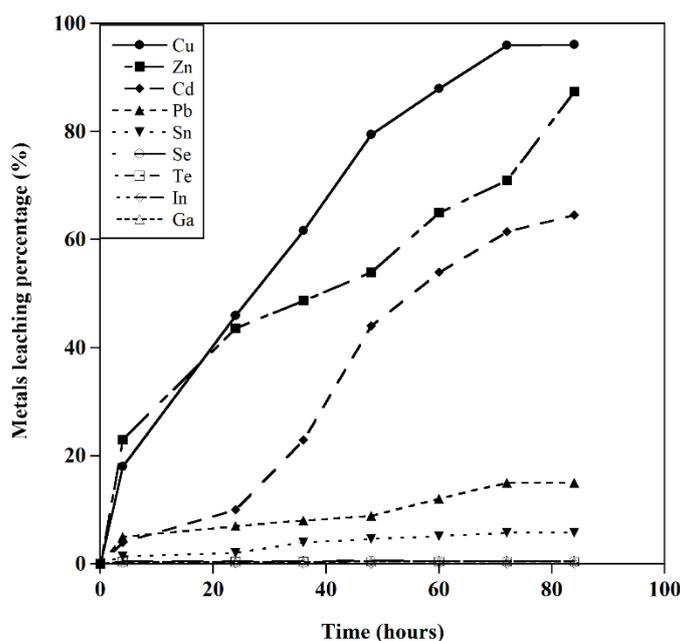
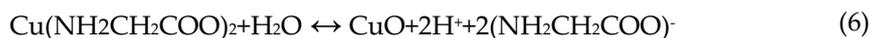


Figure 2. The leaching behavior glycine concentration=0.5 M, particle size $\leq 1\text{ mm}$, $T=25^{\circ}\text{C}$, $\text{S/L} = 20\text{gr/l}$.

3.1.1. Effect of Initial pH

According to reports [77,83,84], glycine formed soluble complexes with both cupric and cuprous ions and equilibrium between copper oxides and copper glycinate as shown in the below equations:



There are different species of glycine in solution at different pH values, as shown in Table 3 [77,85]. The region of stability for the species in the Cu-glycine-water system is shown in Figure 3a. Increasing the initial pH to a certain level is expected to enhance the extraction efficiency of copper.

Table 3. Stability constants of copper glycinate species at 25 °C and 1 atm [77].

Reaction	Stability Constant
$\text{Cu}^{2+} + 2(\text{NH}_2\text{CH}_2\text{COO})^- = \text{Cu}(\text{NH}_2\text{CH}_2\text{COO})_2$	15.64
$\text{Cu}^{2+} + (\text{NH}_2\text{CH}_2\text{COO})^- = \text{Cu}(\text{NH}_2\text{CH}_2\text{COO})^+$	8.57
$\text{Cu}^+ + 2(\text{NH}_2\text{CH}_2\text{COO})^- = [\text{Cu}(\text{NH}_2\text{CH}_2\text{COO})_2]^-$	10.1
$\text{Cu}(\text{NH}_2\text{CH}_2\text{COO})^+ + \text{H}^+ = \text{Cu}(\text{NH}_3\text{CH}_2\text{COO})^{2+}$	2.92
$(\text{NH}_2\text{CH}_2\text{COO})^- + \text{H}^+ = \text{Cu}(\text{NH}_3\text{CH}_2\text{COO})$	9.778
$\text{H}(\text{NH}_2\text{CH}_2\text{COO}) + \text{H}^+ = \text{H}_2(\text{NH}_2\text{CH}_2\text{COO})^+$	2.350

Figure 3b illustrates copper recovery at various initial pH values, at a temperature of 25°C, and a solid/liquid ratio of 20 gr/l. The experiments were conducted with an acid concentration of 0.5 M. Glycine has been found to be an effective leaching agent for copper extraction from other sources, such as ore [86–88] and PCB waste [89–91], as reported in previous studies.

In this step, experiments were conducted with pH values ranging from 8 to 13. As shown in Figure 3b, at a time of 60 hours, increasing the pH from 8 to 10 led to a significant increase in copper extraction yield, from 38% to 90%. However, this trend stopped at pH values above 10 due to the formation of insoluble Cu₂O or CuO according to the Eh-pH diagram [91]. The leaching reaction for copper was controlled by stabilizing the formation of complexes with Cu ions and glycinate anions [91]. The optimized condition for the highest Cu extraction (96.9%) was found to be at pH 10 after 75 hours, as shown in the (Eh-pH) diagram in Figure 3a.

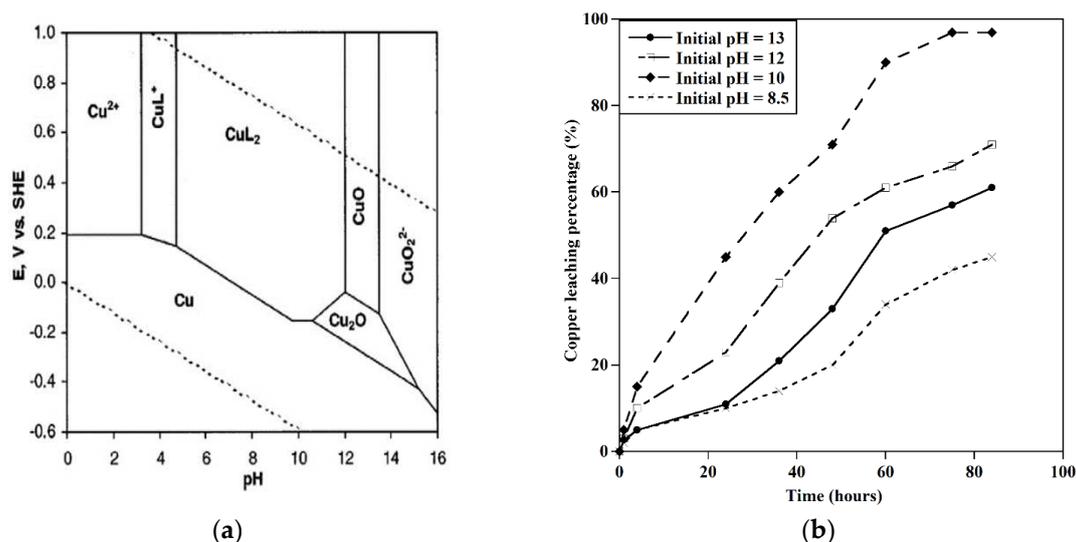


Figure 3. a) Potential/pH diagram for the Cu-water-glycine system at 25 °C and 1 atm[77]. b) Effect of initial pH on the extraction of Cu glycine concentration=0.5 M, S/L: 20 gr/l, particle size=1mm, T=25 °C.

The leaching behavior of other metals was investigated at the optimized conditions for copper extraction. Figure 4a shows that at an initial pH of 10 and a glycine concentration of 0.5 M, zinc extraction achieved about 88% recovery after 72 hours, whereas lead extraction did not exceed 15%. It is expected that Zn^{2+} and Pb^{2+} form different complexes with glycinate anions, which may explain the differences in their extraction rates [92,93]. Although stable lead-glycinate complexes formed at initial pH values above 10, the dissolution rate of lead did not increase significantly. The main point of this step is the selective leaching of copper, zinc, and cadmium over other metals at a pH of 10. As shown in Figure 4b, lead and tin can be dissolved completely by increasing the pH to 13. Based on the leaching behavior of the metals, it is recommended that the dissolution of copper and zinc be carried out at pH 10 in a countercurrent process. In contrast to the other metals, the leaching of cadmium decreased by increasing the initial pH, and its dissolution reached a maximum of 73% at pH levels below 10 by glycine 0.5 M. In the second step, the leaching of lead and tin can be conducted in a countercurrent process at pH 13, achieving extraction rates of 62% and 81.8%, respectively.

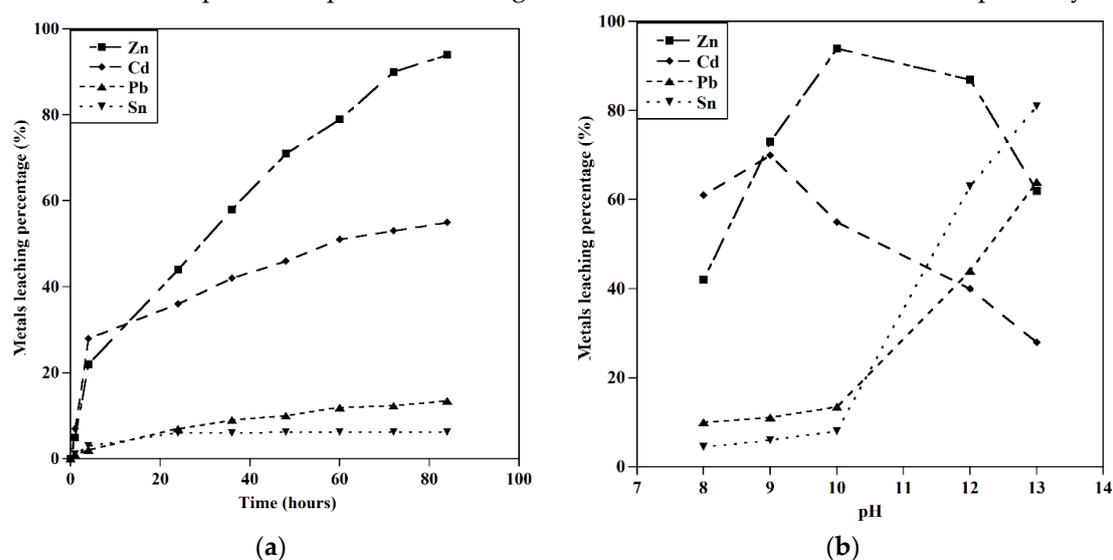


Figure 4. -a) The leaching behavior of metals (Glycine concentration=0.5M, T=25 °C, S/L: 20 gr/l, initial pH= 10, particle size=1mm), b) Effect of pH on the leaching behavior of metals (Glycine concentration=0.5M, T=25 °C, S/L: 20 gr/l, particle size=1mm).

3.1.2. Effect of Glycine Acid Concentration

Figure 5 illustrates copper and lead recovery at various acid concentrations, at a temperature of 25°C and a solid/liquid ratio of 20 gr/l. Experiments were conducted over a range of acid concentrations from 0.1 M to 1.5 M to evaluate the effect of glycine concentration while keeping the initial pH, solid-to-liquid ratio, and temperature constant at pH 10, 20 gr/l, and 25°C, respectively. Based on the glycine-to-copper molar ratio, an increase in acid concentration is expected to increase the extraction yield of copper. As shown in Figure 5, copper recovery was enhanced by increasing the acid concentration from 0.1 M to 1.5 M. However, above acid concentrations of 0.5 M, there was no significant increase in copper extraction. Moreover, after the acid concentration of 0.5 M, the extraction of copper and zinc was decreased. The effect of increasing glycine acid concentration on zinc dissolution behavior is similar to that of copper leaching, and the optimized leaching of zinc is achieved at a 0.5 M glycine solution. The leaching of other metals was investigated at different acid concentrations. Figure 5 shows that increasing the glycine concentration from 0.1 M to 1.5 M had a positive effect on the extraction of lead and cadmium.

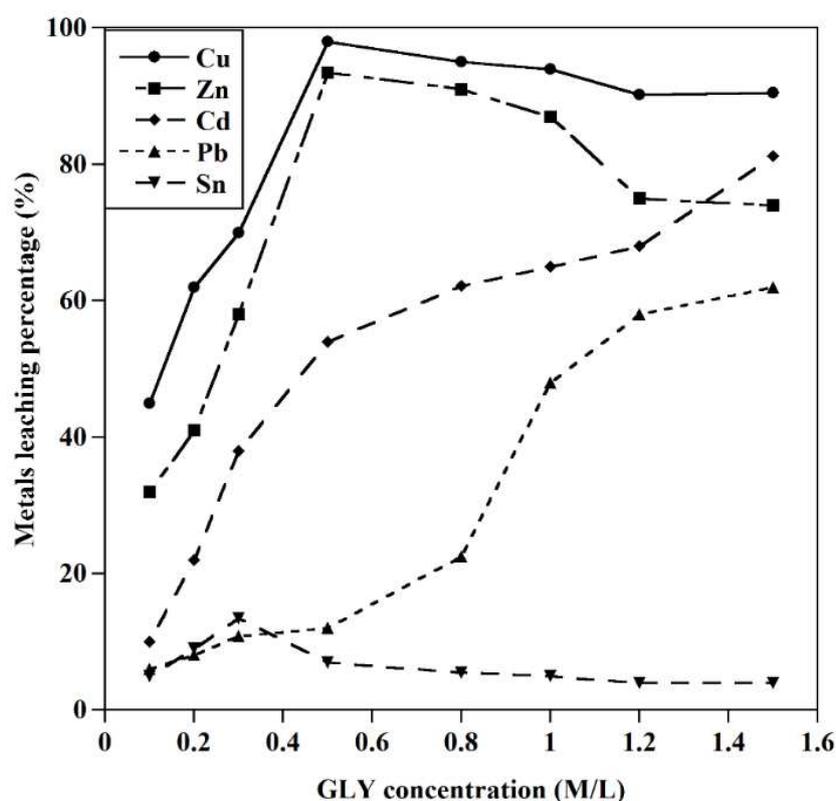


Figure 5. The effect of [GLY] concentration on the leaching behavior of metals (T=25 °C, S/L: 20gr/l, initial pH=10).

3.1.3. Effect of the Solid/Liquid Ratio

The influence of solid/liquid ratio on copper and other base metals recovery was investigated from 5 to 200 gr/l, and the optimized ratio controls the consumption of the leaching agent. The conditions of experiments were kept at the initial pH of 10, the acid concentration of 0.5 M, and 25 °C. As shown in Figure 6, the dissolution of copper will increase when the S/L ratio decreases from 5 gr/l to 200 gr/l. Although, there is no significant difference in the range of 5-20 gr/l. Similar to copper extraction, this decreasing trend is seen in the extraction of other metals if the S/L ratio increases.

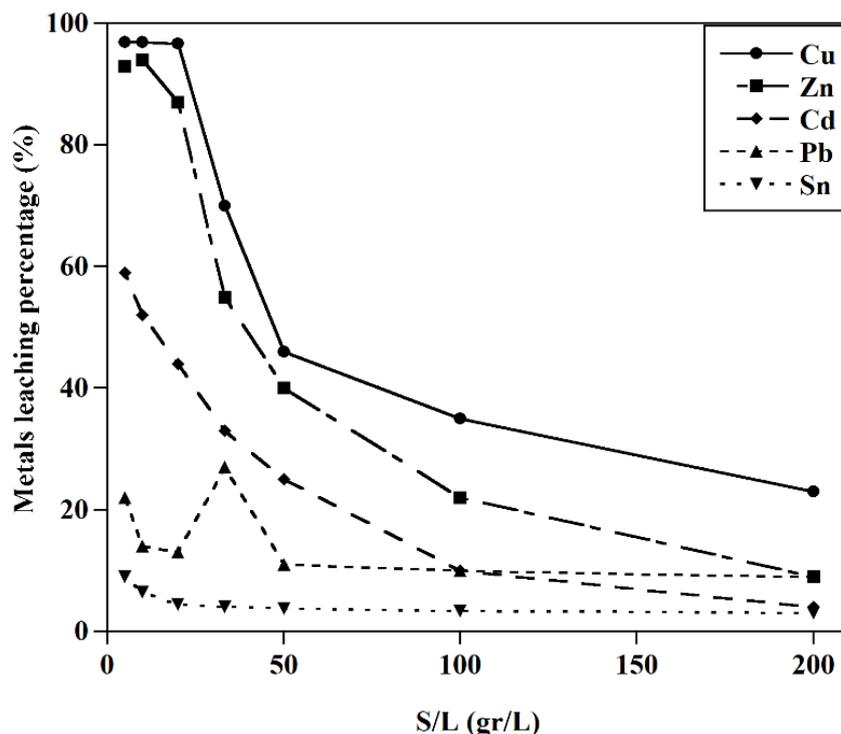


Figure 6. The effect of S/L on the leaching behavior of metals ($T=25\text{ }^{\circ}\text{C}$, $[\text{GLY}]:0.5\text{M}$, initial $\text{pH}=10$).

3.1.4. Effect of Temperature

To investigate the influence of temperature on the extraction of metals, experiments were conducted at an elevated $45\text{ }^{\circ}\text{C}$ temperature at 0.5 M glycine, $\text{S/L}:20\text{ gr/l}$ and 10 initial pH . As shown in Figure 7, the extraction of copper and cadmium increased very little with the increase in temperature. But, the results showed that the increasing of temperature reduced the extraction of lead, zinc, and tin. Due to the decomposition of glycine through deamination and decarboxylation, the pH of the solution can be reduced during the leaching process [84,94]. The decrease in zinc, lead, and tin extraction was due to the decrease in the solution pH . Also, the increase in cadmium extraction could be caused by this phenomenon.

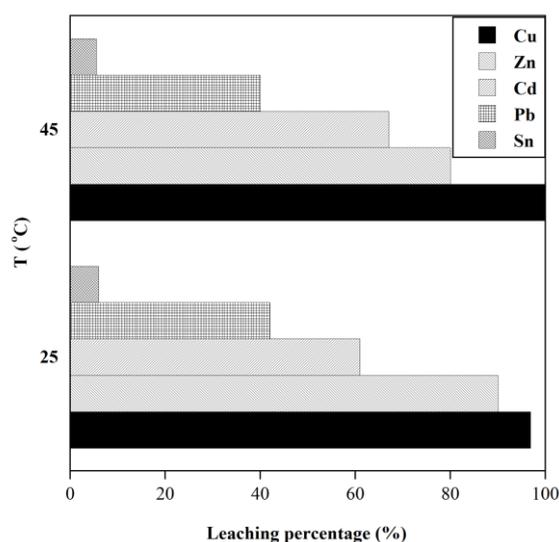


Figure 7. Effect of temperature on the extraction of metals for 72 h ($[\text{GLY}]=0.5\text{ M}$, $\text{S/L}:20\text{ gr/l}$, initial $\text{pH}=10$).

3.1.5. Effect of Hydrogen Peroxide

Hydrogen peroxide (H_2O_2) is used as an oxidizing agent in the leaching procedure, as the dissolution of metals with high reduction potentials is increased by applying a strong oxidant [95]. H_2O_2 is a strong oxidant with a standard electrode potential of 1.83 V, making it useful in the copper leaching system [96,97]. To evaluate the effect of oxidants on the extraction of base metals, experiments with H_2O_2 additions were conducted in comparison with ambient O_2 . The experiments were carried out at a glycine concentration of 0.5 M, an initial pH of 10, a solid-to-liquid ratio of 5 gr/l, room temperature, and solid samples with a particle size of less than 1 mm. Figure 8 shows the extraction of copper over 84 hours of leaching.

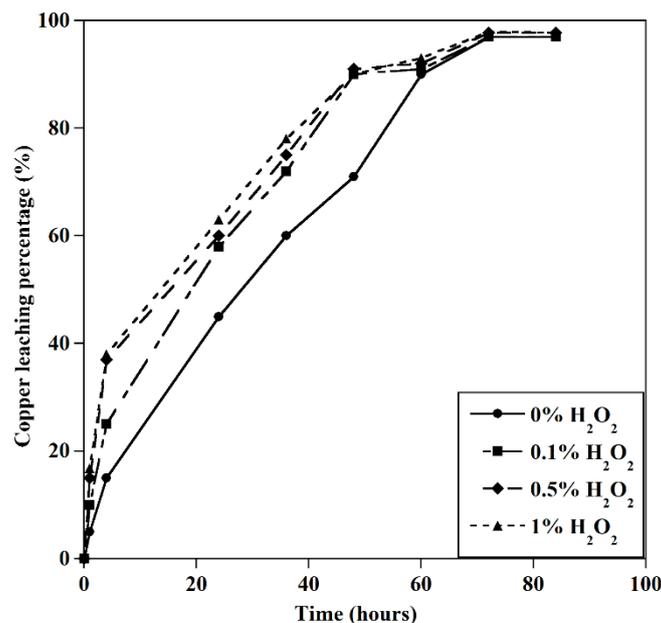


Figure 8. The effect of H_2O_2 on the leaching of copper ($T=25\text{ }^\circ\text{C}$, $[\text{GLY}]=0.5\text{M}$, $\text{S/L}: 5\text{ gr/l}$, initial $\text{pH}=10$).

As expected, the application of H_2O_2 increased the dissolution of copper, particularly in the initial hours of leaching. At the 48-hour mark, the addition of 0.5%-1% H_2O_2 increased the copper extraction from 71% in the O_2 ambient to 90% in the presence of 1% H_2O_2 .

Figure 9 shows that the effects of an increasing H_2O_2 concentration on the extraction of other metals were different. Although the dissolution of zinc, tin, lead and cadmium was slightly improved by the addition of 1% H_2O_2 .

Based on the reports, at high pHs, the generation of hydroxide ions and oxygen happened due to the decomposition of H_2O_2 as shown below equations:



The generated hydrogen radical (OH^\cdot) improved the leaching of cadmium and tin due to the high standard reduction potential. Also, the decomposition of H_2O_2 into O_2 generated an additional oxidant [98].

The insignificant change in lead dissolution with the increase of H_2O_2 dosage was due to a decrease in pH caused by the oxidation of glycine with H_2O_2 [84].

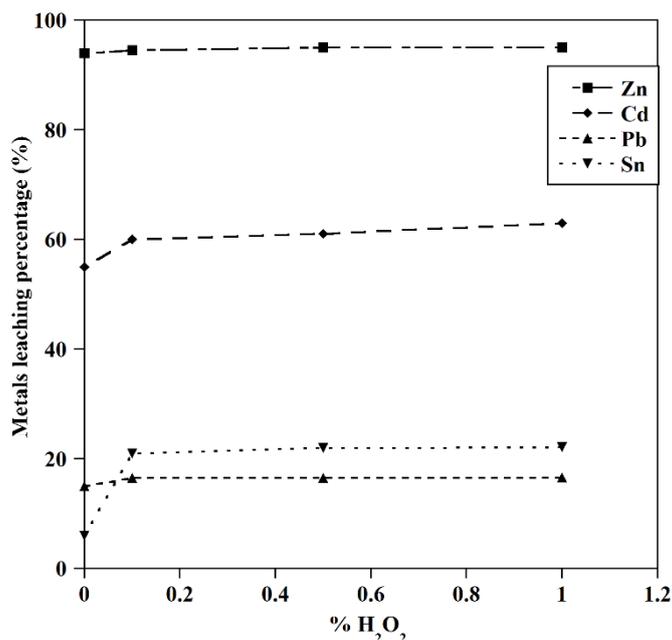


Figure 9. The effect of H₂O₂ on the leaching of metals (T=25 °C, [GLY]=0.5 M, S/L: 5 gr/l, initial pH=10).

3.1.6. The Optimization of the Leaching Condition

The interaction of parameters indicates that the effect generated by changing one variable depends on the level of other variables. The optimized conditions were calculated by analyzing data for the leaching of copper, zinc, cadmium, lead, and tin. Based on the responses and analysis of the variance presented in Table 4, a statistical model using the Box-Behnken model was computed to determine the extraction of all metals. The coefficient of determination (R²), adjusted R-square (adj. R²), and analysis of variance (ANOVA) tests were employed to assess the adequacy of the proposed model and its goodness of fit. The goodness-of-fit statistics for all of the response model are provided in Tables 5 and 6. As reported, the high value in the models was representative of the significant models. Also, as can be observed the p-Values of models are negligible that indicated the proposed models were unity and significant. The determination coefficients were above 0.90 for all metals indicating the appropriate efficiency of the proposed models.

The semi-empirical relation for metals extraction containing interactions between the existing parameters is defined as follows:

$$\begin{aligned} \text{Recovery of Copper (\%)} = & 98.16791 + 92.94 * [\text{GLY}] - 0.94839 * \text{S/L} - 4.83706 * \text{pH} - \\ & 40.3195 * \text{H}_2\text{O}_2 - 46.3501 * [\text{GLY}] * \text{H}_2\text{O}_2 + 8.15275 * \text{pH} * \text{H}_2\text{O}_2 - 40.27973 * [\text{GLY}]^2 + \\ & 0.00307692 * \text{S/L}^2 \end{aligned} \quad (11)$$

$$\begin{aligned} \text{Recovery of Zinc (\%)} = & 40.76864 + 92.97376 * [\text{GLY}] - 1.08869 * \text{S/L} + 95.033 * \text{H}_2\text{O}_2 - \\ & 46.4488 * [\text{GLY}] * \text{H}_2\text{O}_2 - 42.0622 * [\text{GLY}]^2 + 0.003645 * \text{S/L}^2 - 45.95 * \text{H}_2\text{O}_2^2 \end{aligned} \quad (12)$$

$$\begin{aligned} \text{Recovery of Cadmium (\%)} = & 60.17453 + 85.1278 * [\text{GLY}] - 0.975375 * \text{S/L} - 5.04142 * \text{pH} + \\ & 39.085 * \text{H}_2\text{O}_2 - 0.174766 * [\text{GLY}] * \text{S/L} - 28.1142 * [\text{GLY}] * \text{H}_2\text{O}_2 + 0.046859 * \text{S/L} * \text{pH} - \\ & 0.1904 * \text{S/L} * \text{H}_2\text{O}_2 - 19.275 * [\text{GLY}]^2 + 0.002308 * \text{S/L}^2 \end{aligned} \quad (13)$$

$$\begin{aligned} \text{Recovery of Lead (\%)} = & 243.24 - 39.386 * [\text{GLY}] - 0.943714 * \text{S/L} - 43.89817 * \text{pH} + 7.33 * \\ & [\text{GLY}] * \text{pH} + 0.066310 * \text{S/L} * \text{pH} + 0.001021 * \text{S/L}^2 + 2.06491 * \text{pH}^2 \end{aligned} \quad (14)$$

$$\text{Recovery of Tin (\%)} = 104.91353 - 19.30147 * [\text{GLY}] + 1.39876 * \text{S/L} - 39.65141 * \text{pH} + 54.54306 * \text{H}_2\text{O}_2 + 3.86568 * [\text{GLY}] * \text{pH} - 0.143727 * \text{S/L} * \text{pH} - 4.67082 * \text{pH} * \text{H}_2\text{O}_2 - 8.71794 * [\text{GLY}]^2 + 2.88745 * \text{S/L}^2 \quad (15)$$

The positive terms indicate a synergistic effect on the extraction, whereas the negative terms express antagonism. As discussed in the prior section, the effect of parameters was consistent with actual results.

As shown in Figure 10, the leaching condition for copper was plotted by the Minitab software: [GLY]=0.5 M, S/L: 10 gr/l, pH=10, and H₂O₂ 1%. Similar to copper, based on the results of experiments, the optimal leaching conditions for other metals were reported in Table 7.

3.2. The Leaching Behavior of Valuable Metals

The residue from the base metals leaching process was washed with double distilled water, filtered, and then utilized as raw material for the leaching of other metals. The residue from the base metals leaching was roasted at a constant temperature of 400°C for a specified time to achieve phase transformation. Previous studies have identified the first exothermic peak in the DTA curve at 370°C for the oxidation of CIGS at the surface [99,100]. Additionally, the temperature range of 200-400°C has been determined to produce In₂O₃ and Ga₂O₃ [101]. Therefore, a temperature of 400°C was selected for roasting for a duration of 1 hour. Figure 11 illustrates the XRD pattern of the dried and roasted leaching residue after glycine leaching. As predicted, the formation of SeO₂, Ga₂O₃, and In₂O₃ was achieved due to thermal oxidation. The presence of CuIn_{0.5}Ga_{0.5}Se₂ and SnO₂ in the pattern indicates the existence of a few amounts of copper and tin in the sample. The reason is that the glycine leaching experiments before roasting was conducted at the S/L:20 gr/l. Table 8 shows the content of the dried material analyzed after the glycine leaching step. Due to the nature of the existing phases, it appears that indium and gallium form soluble species in the lower pH range. According to the Hard-Soft Acid Base (HSAB) concept, strong acids such as In³⁺ and Ga³⁺ prefer to bind to strong bases such as Cl⁻ to form ionic complexes. Therefore, it is expected that HCl is a suitable leaching agent for the dissolution of indium and gallium [102–104].

Table 4. Response surface design of experiments.

Std	Factor	Factor	Factor	Factor	Respons	Respons	Response	Respons	Respons	
	1	2	3	4	e 1	e 2	3	e 4	e 5	
Ru	A:[B: S/L	C: pH	D:	Recovery	Recovery	Recovery	Recovery	Recovery	
n	GLY]			H ₂ O ₂	of	of Zinc	of	of Lead	of Tin	
No.	M	gr/l		%	Copper		Cadmium			
9	1	0.1	100	10	1	25	20	4.8	1.5	2
16	2	1	20	10	0	92	88	62	44	5.5
20	3	1.5	100	10	0.5	36	22.8	31	51	2.4
2	4	1.5	20	10	0	90	72.5	81	67	5.6
14	5	1	10	10	0	92	88.5	75	57	5
7	6	0.8	100	10	0	28	17	25	15	4.1
15	7	0.8	50	10	0.5	54.6	49.1	37.5	21.5	9.1
4	8	1.5	10	13	0	62	55	72	97.8	98
26	9	1.5	5	9	1	43	55	99	60	5
10	10	1.5	5	10	1	93	75	84	62	6
1	11	0.1	5	10	0	40	30	10	5	5
3	12	0.1	200	10	0.5	12	11	0	0	0

22	13	1.5	5	13	1	65	56	73	99.8	99.7
8	14	0.8	100	13	0	14	11	20	77	54
19	15	0.1	100	13	0.5	21	18	0	16	24
18	16	1.5	100	8	0.5	18	11.5	27	14	7.4
25	17	0.5	100	10	0.5	43	34	17	7	3
27	18	0.8	100	10	0.5	33	25	29.5	15	2
12	19	0.5	20	10	1	98.5	98.1	62	16	5.8
28	20	0.8	100	10	0	28	17	31.7	22	4.1
17	21	0.1	100	8	0.5	16	15	3.2	1	2
23	22	0.8	20	10	1	72	69	29.9	24	10.3
6	23	0.5	20	13	0	67	62	19.5	81.9	64.9
5	24	0.8	50	10	0.5	54.6	49.1	19.5	21.5	9.1
24	25	0.8	200	10	1	36	28	13	21	8
11	26	0.1	100	10	1	22.5	18.1	7.8	7	7
13	27	0.5	10	10	0.5	99	99	60	22	10
21	28	0.5	10	10	1	99.9	99.5	63	22.1	11

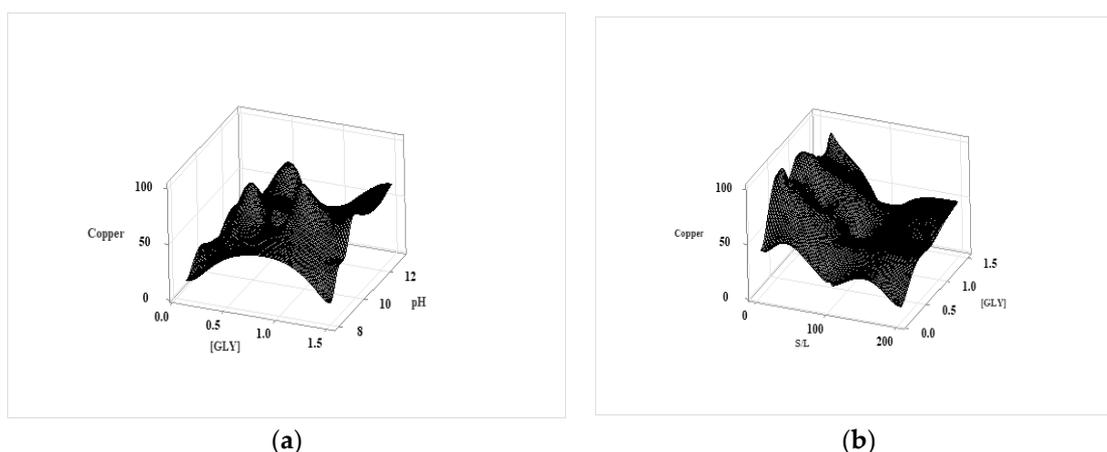


Figure 10. The RSM plot of Cu recovery.

Table 5. ANOVA Table for Recovery of copper recovery for Quadratic and Reduced Quadratic model.

Analyzed Model	Recovery of Copper							
	Quadratic model				Reduced Quadratic model			
	Coefficient	Sum of Squares	F-value	p-value	Coefficient	Sum of Squares	F-value	p-value
Intercept	38.64				98.09			
[GLY]	3.99	78.68	0.40	0.5404	96.49	109.83	0.64	0.4346
S/L	-31.18	3,101.11	15.58	0.0017	-0.96	6,072.01	35.23	< 0.0001

pH	-0.45	0.51	0.0 0	0.960 6	-4.93	36.40	0.21	0.651 0
H ₂ O ₂	1.27	7.70	0.0 4	0.847 1	-36.97	210.62	1.22	0.282 8
[GLY] * S/L	3.85	29.53	0.1 5	0.706 4				
[GLY] * pH	-7.75	178.75	0.9 0	0.360 6				
[GLY] * H ₂ O ₂	-20.29	1,826.81	9.1 8	0.009 7	-49.71	2,240.58	13.00	0.001 9
S/L * pH	-7.61	42.40	0.2 1	0.652 1				
S/L * H ₂ O ₂	-4.01	40.75	0.2 0	0.658 4				
pH * H ₂ O ₂	8.70	235.23	1.1 8	0.296 8	8.01	407.07	2.36	0.140 8
[GLY] ²	-17.66	1,056.01	5.3 1	0.038 4	-42.05	2,432.72	14.12	0.001 3
S/L ²	35.90	2,183.67	10. 97	0.005 6	0.00	3,282.95	19.05	0.000 3
pH ²	-7.81	104.79	0.5 3	0.481 0				
H ₂ O ₂ ²	-8.74	322.93	1.6 2	0.225 1				
Model summary		21,697.64	7.7 9	0.000 3		21,010.8 5	15.24	< 0.000 1
		Significant			Significant			
Residual Lack of Fit		2,587.76	-	-		3,274.55	-	-
		Not Significant*			Not Significant*			

*Based on analysis of the Externally Studentized Residuals vs Normal % Probability graphs.

Table 6. Fit statistics for the suggested models.

	Model	R ²	Adjusted R ²	Predicted R ²	ANOVA p- value
Recovery of Copper	Quadratic model	0.893	0.779	-0.674	0.0003
	Reduced Quadratic model	0.865	0.808	0.576	<0.0001
Recovery of Zinc	Quadratic model	0.928	0.851	-0.040	<0.0001
	Reduced Quadratic model	0.909	0.878	0.811	<0.0001
	Quadratic model	0.920	0.834	0.316	<0.0001

Recovery of Cadmium	Reduced Quadratic model	0.902	0.860	0.800	<0.0001
Recovery of Lead	2FI model	0.926	0.883	0.313	<0.0001
	Reduced 2FI model	0.906	0.879	0.670	<0.0001
Recovery of Tin	Quadratic model	0.980	0.958	0.632	<0.0001
	Reduced Quadratic model	0.967	0.958	0.831	<0.0001

Note: R^2 represents the coefficient of determination, adjusted R^2 represents the adjusted coefficient of determination, Predicted R^2 represents the predicted coefficient of determination, and ANOVA p-value indicates the statistical significance of the model fit.

Table 7. The optimal Conditions for the leaching of metals.

	[GLY](M)	S/L (gr/l)	pH	H ₂ O ₂ (%)	Recovery (%)
Copper	0.5	10	10	1	99.9
Zinc	0.5	10	10	1	99.5
Cadmium	1.5	5	9	1	99
Lead	1.5	5	13	1	99.8
Tin	1.5	5	13	1	99.7

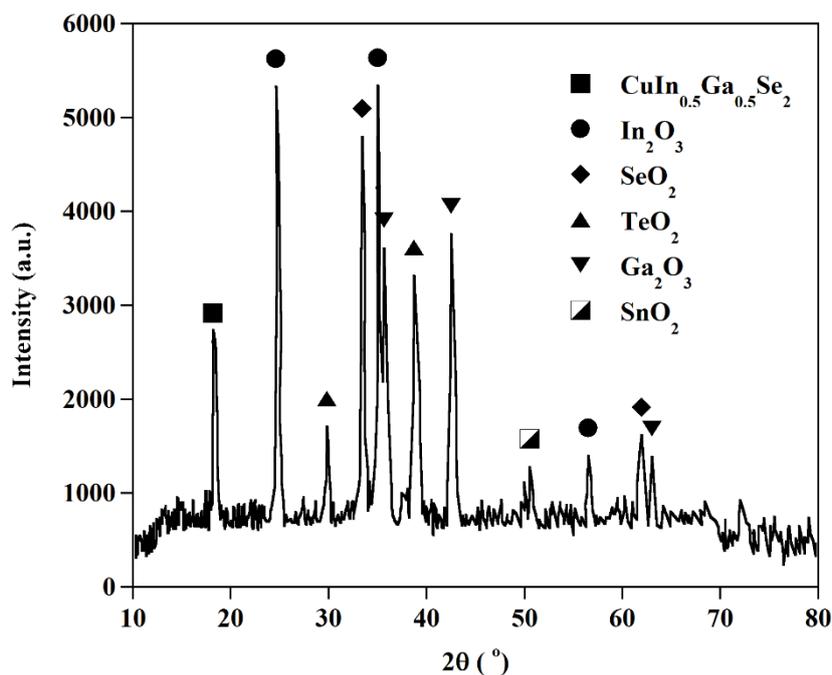


Figure 11. XRD pattern of glycine leaching residue after oxidation roasting.

Table 8. The XRF results of major metal contents of glycine leaching residue (feed material of HCl leaching).

	Cu	Pb	Sn	Se	In	Ga	Te
Metal content (Wt%)	5	1.1	3.9	23.2	10.4	9.8	25

Figure 12 illustrates the recovery of In, Ga, Se, and Te under the following leaching conditions: [HCl]: 4 M, T=25°C, S/L:10 gr/l. As shown in Figure 13, the leaching efficiencies for gallium and

indium increased as a function of time. As predicted, the recovery of indium and gallium was 93% and 71%, respectively, and became saturated at 100 min. Selenium and tellurium were recovered at 21.8% and 11.5%, respectively, as shown in Figure 13. However, a significant amount of tellurium and selenium was not dissolved, which could be due to the lack of soluble species of selenium and tellurium in this range of concentration.

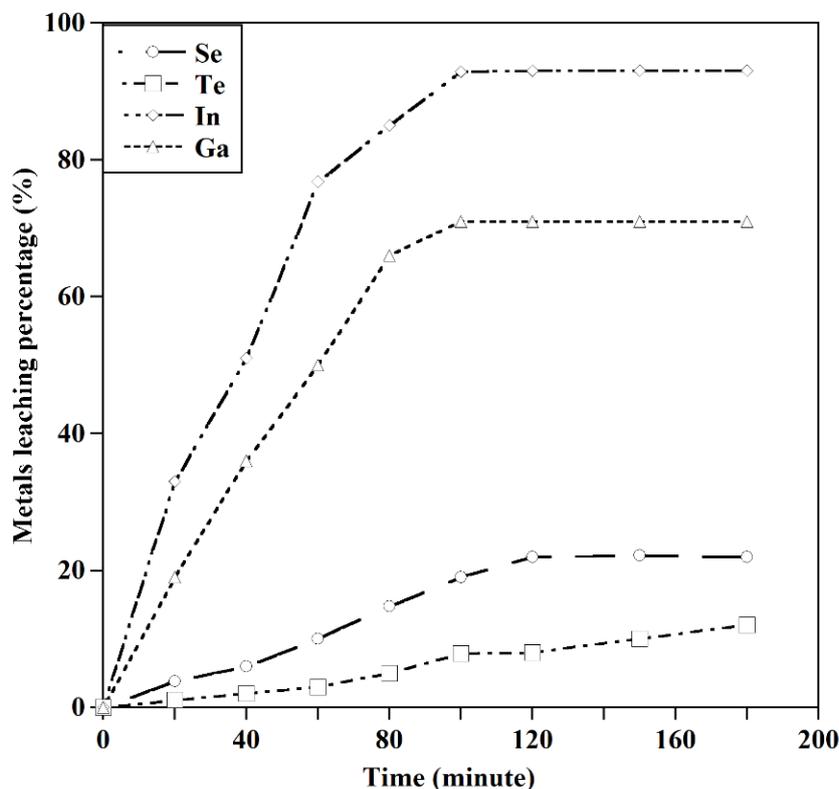


Figure 12. The leaching behavior of In, Ga, Se, and Te ([HCL]:4 M, T=25°C, S/L:10 gr/l).

3.2.1. Effect of HCl Concentration

The effect of HCl concentration on the leaching efficiencies of the sample was investigated, with acid concentrations ranging from 0.5 M to 4 M. As shown in Figures 13a,b, the recovery of indium and gallium increased with increasing acid concentration. When the acid concentration increased from 0.5 M to 5 M, the leaching efficiency of indium and gallium increased from 56% to 97% and from 21% to 85%, respectively. However, as shown in Figure 13d, the leaching of selenium decreased with an increase in acid concentration. This issue could be related to the range of stability of Se species. Soluble species, including H_2SeO_3 and HSeO_4^- , exist at acidic pH values [105]. However, by decreasing pH values, selenium precipitates as elemental Se. Thus, increasing the acid concentration promotes the precipitation of selenium and decreases its leaching efficiency.

The leaching percentage of selenium increased from 21.8% to 56.7% by reducing the acid concentration from 5 M to 0.5 M. However, acid concentration's effect on tellurium's recovery was more complicated. Te^{4+} exists in the form of ions such as $\text{Te}(\text{OH})_3^+$ or $\text{TeO}(\text{OH})^+$ in very low pH ranges. As seen in Figure 13c, the highest recovery percentage of tellurium (37.5%) was obtained at an acid concentration of 1 M. This could be due to the narrow region of soluble $\text{Te}(\text{OH})_3^+$ species at low pH values [106], which results in a low leaching rate of tellurium under these conditions.

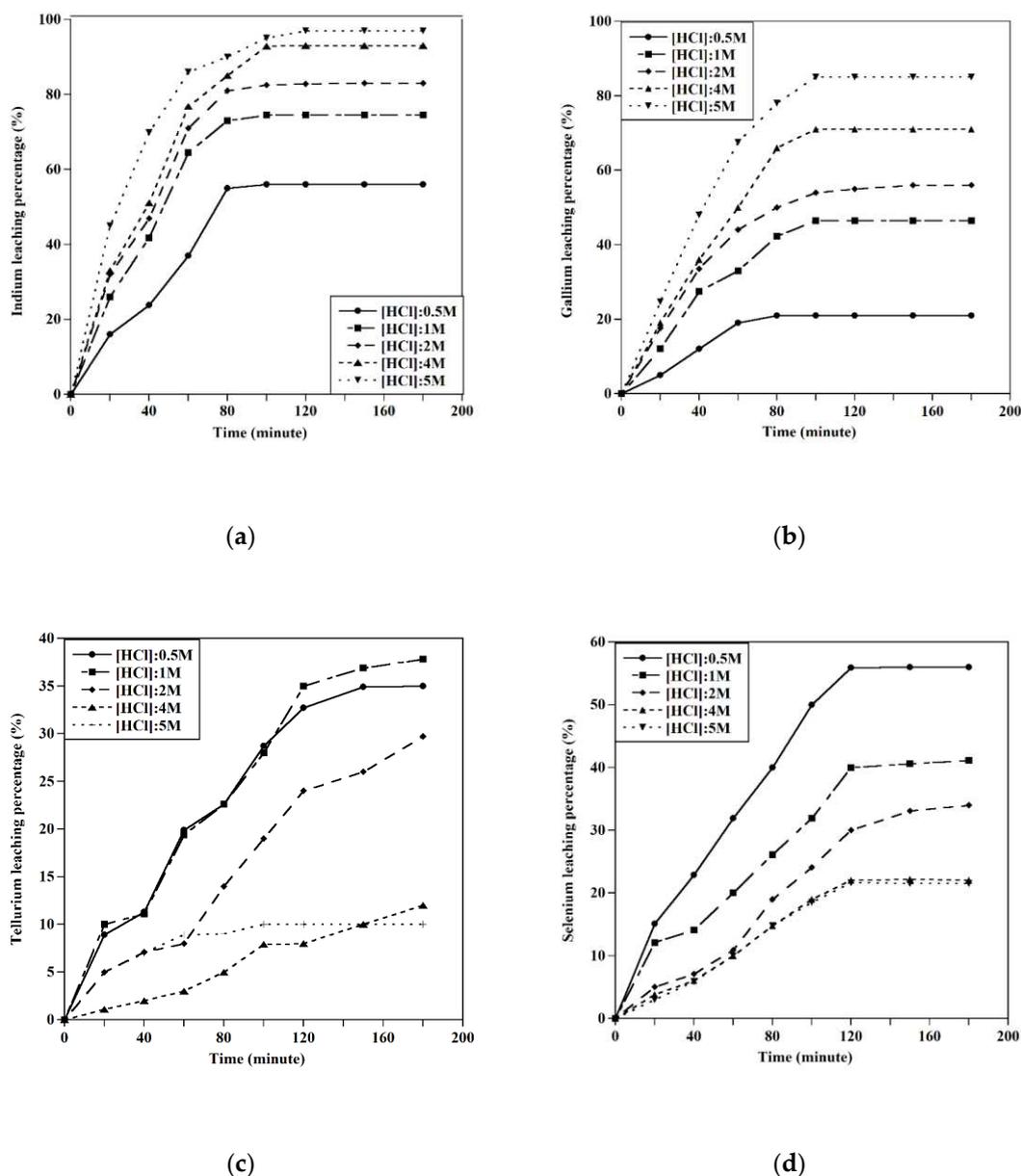


Figure 13. The effect of [HCl] concentration on the leaching behavior of metals ($T=25\text{ }^{\circ}\text{C}$, $S/L:10\text{ gr/l}$)
a) Leaching of indium, b) Leaching of gallium, c) Leaching of tellurium, and d) Leaching of selenium.

3.2.2. Effect of S/L Ratio

The effect of solid/liquid ratio on indium, gallium, selenium, and tellurium recovery was investigated from 5 to 200 gr/l. The conditions of experiments were kept at the acid concentration of 5 M, and $25\text{ }^{\circ}\text{C}$. As shown in Figure 14, the dissolution of copper will increase when the S/L ratio decreases from 5 gr/l to 200 gr/l. As was predictable, the decreasing trend is seen in the extraction of other metals if the S/L ratio increases.

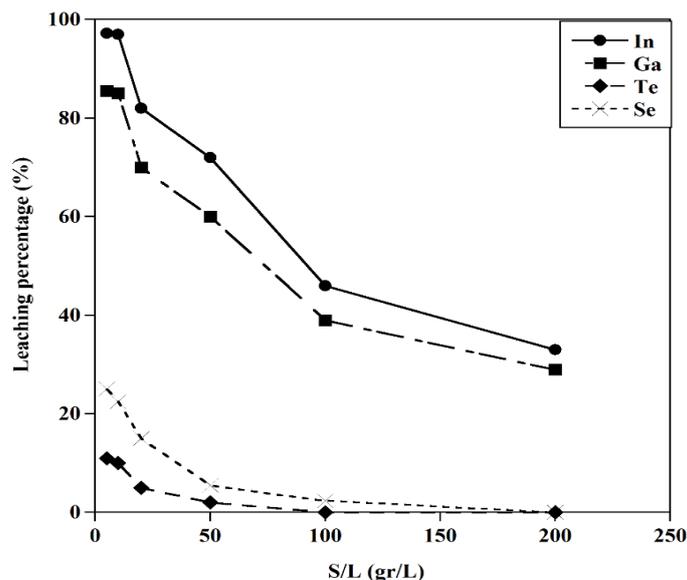


Figure 14. The effect of S/L on the leaching behavior of metals ($T=25\text{ }^{\circ}\text{C}$, $[\text{HCl}]=5\text{M}$, $t=120\text{ min}$).

3.2.3. Effect of Temperature

The effect of temperature on the extraction of metals was studied at the range of $25\text{--}60\text{ }^{\circ}\text{C}$. According to the optimal condition for the In, Ga, Se, and Te recovery, two different conditions were considered. As shown in Figure 15a, the HCl concentration was 5M for the high extraction of indium and gallium at the range of temperature $25\text{--}45\text{ }^{\circ}\text{C}$. The results indicated the increasing temperature improved the extraction of indium and gallium to about 100%. Considering the second condition at the $[\text{HCl}]$ of 0.5 M, the extraction of selenium increased by the increasing of temperature until $60\text{ }^{\circ}\text{C}$. On the contrary, the increasing temperature reduced the extraction of tellurium. This can be caused due to the narrow region of tellurium species at elevated temperatures. This decreasing trend of tellurium extraction by the increase of temperature was seen in the sulfuric acid solution [107]. As shown in Figure 15b, the increasing temperature has a positive influence on the indium and gallium extraction at different acid concentrations. The selenium was recovered to about 100% at a temperature of $60\text{ }^{\circ}\text{C}$, whereas the tellurium extraction at this temperature was achieved at less than 10%.

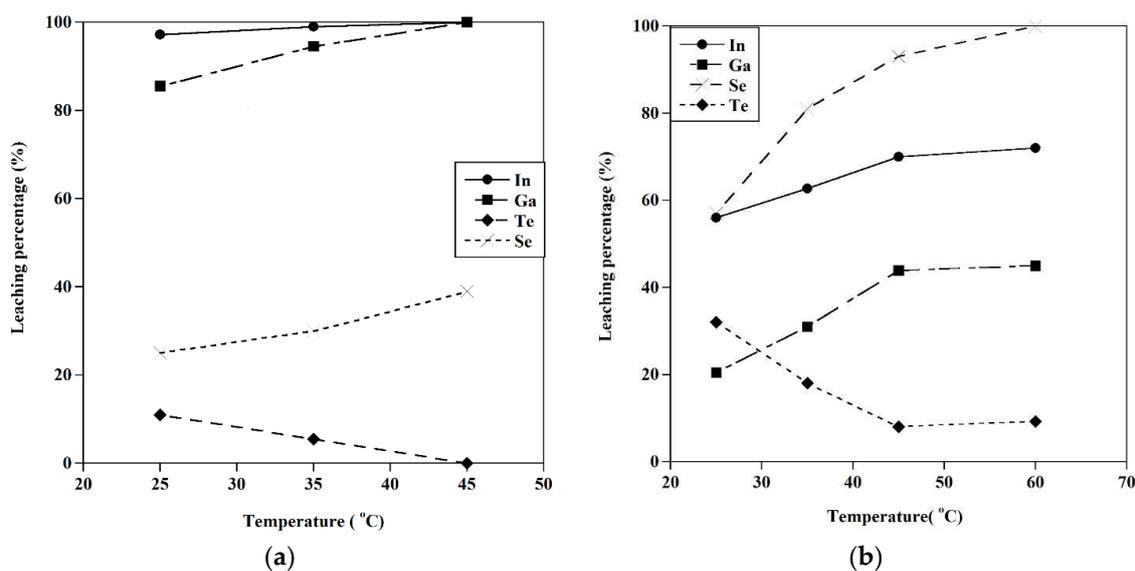


Figure 15. The effect of temperature on the metal extraction a) $[\text{HCl}]=5\text{M}$, $S/L=10\text{ gr/l}$, b) $[\text{HCl}]=0.5\text{ M}$, $S/L=10\text{ gr/l}$, $t=120\text{ min}$.

4. Suggested Flowsheet for Recycling All Types of Solar Panels

Figure 16 represents an innovative and general flowsheet for the recycling of metals from all types of solar panels. The separation of the metallic and non-metallic parts was confirmed by the physical separation, shredding, and thermal treatment. As discussed, the silver extraction was conducted by diluted nitric acid in the first leaching step. Based on this figure, the glycinate solution was used for the leaching of copper, zinc, and cadmium in one step, and other metals such as lead, tin, and the residue of cadmium were dissolved by a 1.5 M glycine solution in the second step. The raffinate which includes zinc, cadmium, and copper, will be processed in our research by solvent extraction method. Additionally, the extraction of other metals such as lead, tin, and cadmium reached their optimized condition by changing the factors. The recovery of indium, gallium, tellurium, and selenium was examined under different conditions, and HCl was used as a leaching agent for the dissolution of indium and gallium. The interesting point was that the separation of tellurium and selenium was conducted by 0.5 HCl at a temperature of 60 °C and a time of 120 min. Also, the indium and gallium were recovered about 100% by 5 HCl at a temperature of 45 °C.

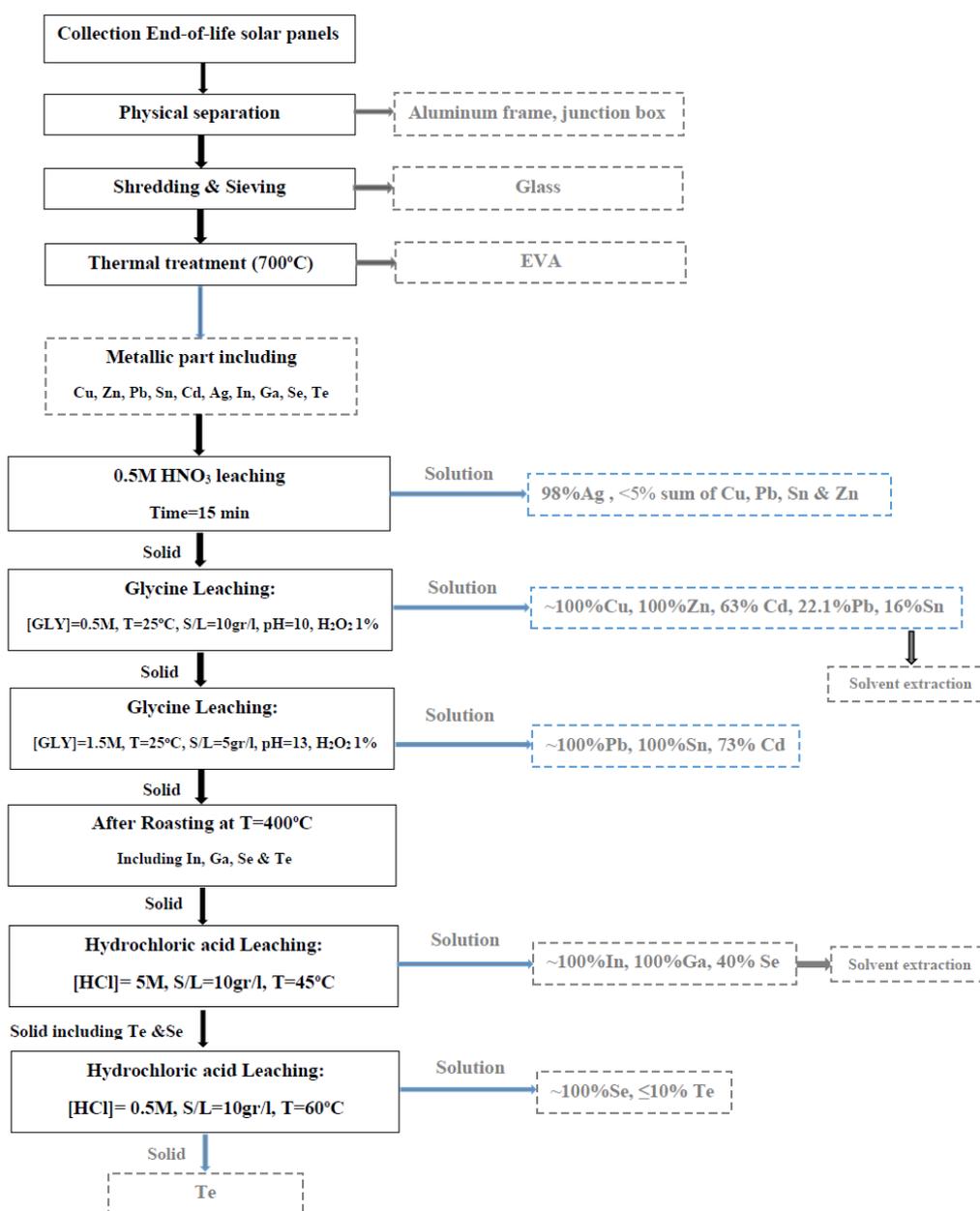


Figure 16. The general flowsheet for recycling all types of solar panels.

5. Conclusions

Life cycle assessment is used to evaluate the environmental effects of solar panel technology. To ensure the effectiveness of solar PV technology, it is necessary to consider all environmental consequences of the significant growth of solar PV production. Comprehensive methods should be developed to recycle spent solar panels after their end of life to protect the environment and achieve economic added value. Advanced research has been initiated to recover metals from various types of solar panels, and this paper focuses on the leaching of base metals, which is the initial step of future research in this field.

This study presents an innovative approach for extracting metals (Cu, Ag, Cd, Te, Se, In, Ga, Sn, Pb, Zn) from all kinds of solar panels. The base metals extraction was a prerequisite for recovering precious metals from end-of-life solar panels. The initial step after preparation was the dissolution of silver using a 0.5 M HNO₃ solution. The leaching behavior of copper and other metals in an alkaline glycine solution was then studied under various conditions. The optimal conditions for the selective extraction of copper and zinc were found to be a glycine concentration of 0.5M, an S/L ratio of 10 gr/l, and an initial pH value of 10. To achieve the highest extraction of other metals (Cd, Sn, Pb), the leaching procedure was performed at different initial pHs, and the optimized conditions for the extraction of Cu, Pb, Cd, Zn, and Sn were studied.

In the final step, the dissolution of indium, gallium, selenium, and tellurium was studied using HCl acid under different conditions. The indium and gallium were recovered at experimental conditions of [HCl]: 5M, T=45°C, and S/L: 10gr/l, achieving recoveries of about 100. By decreasing the acid concentration to 0.5M, the extraction of selenium was conducted at about 100% at a temperature of 60 °C, and completely, the separation of tellurium and selenium happened.

This study demonstrates an innovative approach for extracting metals from all types of solar panels. A comprehensive method for recycling spent solar panels must be developed to ensure the effectiveness of solar PV technology and reduce its environmental impact. The research on recovering metals from different types of solar panels is ongoing, and this paper presents the leaching step of metals, paving the way for future research in this field. Our hope is by conducting kinetic studies, the time of experiments can be optimized. Also, by controlling pH and temperature during the glycine leaching process, we can improve the efficiency of the leaching process. The solvent extraction method can separate copper, Zinc, and cadmium from the glycine solution and indium and gallium from the final solution.

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Conflicts of Interest: The authors declare no conflicts of interest.

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